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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/GB97/01449 <b>(22) International Filing Date:</b> 28 May 1997 (28.05.97) <b>(30) Priority Data:</b> 9611063.0                      28 May 1996 (28.05.96)                      GB <b>(71) Applicant (for all designated States except US):</b> WARWICK INTERNATIONAL GROUP LIMITED [GB/GB]; Mostyn, Holywell CH8 9HE (GB). <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> DAY, Michael, John [GB/GB]; 46 Greendale Road, Port Sunlight, Wirral, Merseyside L62 5DF (GB). <b>(74) Agent:</b> GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> CONCENTRATED ALKALINE ISOTROPIC DETERGENT LIQUID WITH BLEACH <b>(57) Abstract</b> <p>A concentrated alkaline isotropic fabric washing liquid detergent composition comprising at least 23 % of surfactants and further comprising at least 0.2 % hydrogen peroxide bleach, the composition is substantially free from hydrotropes which cause the pH to reduce and impair the effectiveness of the composition. Also a concentrated alkaline isotropic fabric washing liquid detergent composition comprising at least 15 % by weight of surfactant further comprises at least 0.2 % by weight hydrogen peroxide bleach and an optical brightener and is also substantially free from hydrotropes. The compositions may be used as a main wash bleaching liquid or a pretreatment liquid and may be activated with a bleach activator.</p>		

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CONCENTRATED ALKALINE ISOTROPIC  
DETERGENT LIQUID WITH BLEACH

5     This invention relates to concentrated alkaline isotropic fabric washing liquid detergents with hydrogen peroxide bleach.

10     Concentrated alkaline isotropic fabric washing liquid detergents usually contain a hydrotrope to allow a high surfactant level to be used without the viscosity becoming too great. Typical hydrotropes are sodium xylene sulphonate, ethanol, and propylene glycol. International application PCT/GB96 00642 contains such compositions comprising a mixture of nonionic surfactant and anionic surfactant together with a hydrotrope and hydrogen peroxide characterised in that the composition comprises greater than 40% total surfactant when nonionic rich or greater than 20% total surfactant when  
15     anionic rich. A concentrated peroxide liquid is desirable because of the reduced transport and storage costs. A solution to the problem of how to formulate stable concentrated alkaline isotropic fabric washing liquid detergents with peroxide has eluded those skilled in the art for many years.

20     Although there are many publications of concentrated alkaline isotropic fabric washing liquid detergent formulations containing a hydrotrope, there are very few indeed that disclose hydrotrope-free formulations. The closest prior art is probably the 15% anionic (secondary alkane sulphonate) and 3.75% nonionic (primary alcohol ethoxylate (7EO)) composition disclosed in WO96/01311. This formulation had a total surfactant level below 20%. Furthermore, this composition did not  
25     contain additional ingredients such as optical brightener, anti-redeposition or anti-dye transfer agent which renders the composition unsuitable for use as a practical concentrated alkaline isotropic fabric washing liquid detergent with hydrogen peroxide bleach.

30     Thickened peroxide compositions are also known. These contain a thickening system to boost viscosity when only low levels of surfactant are present. GB 2255507 is typical and discloses a

thickened peroxide-containing composition with less than 10% surfactant. These compositions are not suitable for use as laundry detergents. They are thickened in order to give the desired result of a squeezable composition that does not run when put on to a vertical surface. Such compositions do not contain a hydrotrope and always contain less than 10% total surfactant. A skilled person would not use these thickened hydrotrope-free compositions as a starting point for the formulation of a new concentrated fabric washing detergent.

We have now found that the presence of a hydrotrope has a large and very undesirable negative effect on the storage stability of concentrated alkaline isotropic fabric washing liquid detergents, particularly we have found that this is a problem for compositions containing high levels of surfactant. This is a problem for the formulation of concentrated liquid detergents because conventionally such detergents contain hydrotropes. In this specification concentrated means that the liquid in its undiluted state contains more than 15% and preferably at least 20% and most preferably at least 23% total surfactant. Unless stated otherwise all percentages are on a w/w basis.

According to the present invention there is provided a concentrated alkaline isotropic fabric washing liquid detergent composition comprising a total surfactant level which includes at least 15% surfactants and further comprises at least 0.2% hydrogen peroxide bleach and an optical brightener, characterised in that the composition is substantially free from hydrotropes. It is preferred that the composition has no added hydrotrope whatsoever. A small presence of up to 2.5% preferably less than 1.5%, and most preferably less than 0.5% of a material which may function as a hydrotrope may be present perhaps as a result of one of the ingredients of the composition containing this material as a minor constituent or impurity. Such ingredients are advantageously avoided as they probably compromise the shelf life of the composition, however, for an otherwise very stable composition the loss in performance due to a very small amount of material which may function as a hydrotrope may still leave an acceptable shelf life. Such compositions which are substantially free from material which may function as a hydrotrope are therefore within the scope of the present invention.

The total surfactant level is preferably at least 20%, more preferably at least 23 %. Preferably the composition comprises anionic and nonionic surfactants.

5 Any optical brightener that is compatible with peroxide may be used. An optical brightener that has good compatibility with peroxide bleach is Tinopal CBS-X a distyryl biphenyl derivative ex Ciba Geigy. Stilbene based fluorescers may also be used as the optical brightener.

10 According to a second aspect of the invention there is provided a concentrated alkaline isotropic fabric washing liquid detergent composition comprising a total surfactant level which includes at least 23% of surfactants selected from the group comprising anionic and nonionic surfactants, and further comprises at least 0.2% hydrogen peroxide bleach, characterised in that the composition is substantially free from hydrotropes. The composition may also comprise an optical brightener as  
15 defined above.

When present, the nonionic surfactant preferably comprises more than 5% by weight of the total composition and advantageously the composition includes no more than 5% of surfactants which  
20 do not satisfy the pH criteria when tested according to Test A as described hereinafter. Most advantageously it comprises no more than 5% of surfactants which do not satisfy the residual peroxide criteria when tested according to Test A as described hereinafter.

25 The surfactant system is preferably a mixture of anionic and nonionic surfactants, although entirely nonionic or anionic surfactant systems may be used. Compositions may contain an appropriate builder or be unbuilt.

Among the preferred anionic surfactants are:

a) Secondary alkane sulphonates, such as Hostapur SAS 93 a 93% active flake ex Hoechst or Marlon PS65 a sodium n-alkane ( $C_{13-17}$ ) sulphonate with low sodium sulphate content ex Huls;

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b) Linear alkyl ( $C_{10-13}$ ) benzene sulphonic acids such as Marlon AS3 ex Huls;

c) Alkyl ether sulphates such as Neopon LOS70, a 70% active sodium salt ex Witco; and

10 d) Olefin sulphonates such as Hostapur OS supplied as 40% actives ex Hoechst.

Among the preferred nonionic surfactants are:

e) Primary alcohol ( $C_{13-15}$ ) ethoxylates, ethoxylated with from 5 to 9 moles of ethylene oxide, preferably 7 moles, such as Symperonic A7 ex ICI / Cargo Fleet.

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The pH of the composition may lie in the range 7 to 12, and is preferably around 9.5.

The peroxide is preferably present as dissolved hydrogen peroxide in an amount by weight in the range 0.2 to 15% preferably 1 to 8% and most preferably around 5%.

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The composition may also comprise minor components conventionally found in a heavy duty liquid detergent composition. These include perfume, suitably protected enzymes, surfactant preservatives, thickeners, colorants, builders, anti-redeposition agents, anti-dye transfer agents - polyvinylpyrrolidone (PVP) which acts both as an anti-redeposition agent and an anti-dye transfer polymer is preferred - and other conventional additives.

25

The composition preferably contains sequestrants. Preferred sequestrants are sodium diethylene triamine pentamethylene phosphonate, sold as Dequest 2066 by Monsanto, either alone or used with

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2,2' dipyridylamine (DPA) or 1,2'-diaminocyclohexyl tetrakis methylene phosphonic acid. Other sequestering agents for transition metal ions may be used instead of, or in addition to, these preferred sequestrants.

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In a preferred embodiment the compositions according to the invention are activated by use of a bleach activator such as TAED which may be brought into contact with the composition at the time of use by means of a two compartment dispensing system.

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According to a third aspect of the invention the concentrated alkaline isotropic fabric washing liquid detergent composition may be used to pre-treat stained or other specific fabric localities before the fabric is washed. In this process the stain removal is superior to that which can be obtained from the known acid peroxide compositions and the overall performance is enhanced by the high level of surfactant which is possible in compositions according to the present invention. The use of a concentrated formulation also confers the advantage of lower transport and packaging cost, which is environmentally desirable. The pre-treatment method consists of administering the composition according to any one of claims 1 to 11 to a selected area of the item, leaving it in contact with the item for a short treatment period and then washing the item. The treatment period will typically be 1 to 5 minutes, preferably about 2 minutes.

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#### Compatibility of Surfactants with Peroxide

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The following procedure, referred to as Test A is used for identifying suitable surfactants on the basis of pH and peroxide stability data. A trial formulation is prepared using 20% w/w (except Marlinat CM20 which was tested at 15% w/w) of the surfactant to be screened. After sequestration with Dequest 2066, hydrogen peroxide sufficient to give a 5% w/w concentration of peroxide is added and the pH is adjusted to 9.5 by the addition of pre-sequestered caustic solution. Details of the surfactants tested are given in Table 1.

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Table 1 - Surfactants tested

Code	Surfactant	Source	Type	Actives wt%
	Anionic			
A1	Marlon AS3	Huls	linear alkyl (C <sub>10-13</sub> ) benzene sulphonic acid	97
A2	Marlon AS350	Huls	linear alkyl (C <sub>10-13</sub> ) benzene sulphonate, sodium salt	50
A3	Marlon PS65	Huls	secondary alkane (C <sub>13-17</sub> ) sulphonate, sodium salt	65
A4	Hostapur SAS93	Hoechst	secondary alkane (C <sub>13-18</sub> ) sulphonate	93
A5	Empicol 0045	Albright	alkyl sulphate sodium lauryl sulphate	95
A6	Neopon LOS 70	Witco	alkyl ether sulphate lauryl ether sulphate, sodium salt	70
A7	Marinat CM20	Huls	alkyl ether carboxylic acid C <sub>12</sub> /C <sub>14</sub> fatty alcohol polyglycoether (2EO) carboxylic acid	90
A8	Crodasimic LS30	Croda	sarcosinate - RCONCH <sub>2</sub> (CH <sub>3</sub> )COOH, R=alkyl sodium lauroyl sarcosine	30
A9	Adimol CT95	Croda	taurate - RCON(CH <sub>3</sub> )CH <sub>2</sub> CH <sub>2</sub> SO <sub>3</sub> Na, R=alkyl sodium methyl cocoyl taurate	95
A10	Hostapur OS	Hoechst	Olefin sulphonate	40
	Nonionic			
N1	Synperonic A7	ICI	primary alcohol ethoxylate C <sub>13</sub> /C <sub>15</sub> primary alcohol ethoxylate (7EO)	90
N2	Genaminox LA	Hoechst	amine oxide lauryl dimethylamine oxide	30
	Amphoteric			
P1	Dehyton AB30	Henkel	betaine - RN(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> COO, R= alkyl coco-betaine	30

From experience we have derived the following pH and Peroxide Stability Targets. Residual peroxide is determined by permanganate titration. A surfactant should pass both the pH and the peroxide targets. It is possible to use small (<5% preferred) amounts of surfactant which does not pass all the targets, but this is only practical when the majority of the surfactants exceed the targets by a good margin. As will be seen from the test results in Table 2 it is possible to have satisfactory room temperature stability even if the elevated temperature stability is poor. Such surfactants would not be suitable for use in compositions destined to be stored at temperatures in excess of 30°C, which is often the case in warmer climates.

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pH targets:

$\text{pH}_{12}^{25}$	$\geq 9.0$ after 12 weeks at 25°C
$\text{pH}_{12}^{37}$	$\geq 7.5$ after 12 weeks at 37°C
$\text{pH}_4^{45}$	$\geq 8.0$ after 4 weeks at 45°C

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Residual Peroxide targets:

$\text{Per}_{12}^{25}$	$\geq 85\%$ after 12 weeks at 25°C
$\text{Per}_{12}^{37}$	$\geq 50\%$ after 12 weeks at 37°C
$\text{Per}_4^{45}$	$\geq 55\%$ after 4 weeks at 45°C

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Table 2 - Results of Test A

Surfactant	pH <sup>25</sup> <sub>12</sub>	pH <sup>37</sup> <sub>12</sub>	pH <sup>45</sup> <sub>4</sub>	Per <sup>25</sup> <sub>12</sub>	Per <sup>37</sup> <sub>12</sub>	Per <sup>45</sup> <sub>4</sub>
Target	≥9.0	≥7.5	≥8.0	≥85.0	≥50.0	≥55.0
A1	9.7	9.4	9.5	85.0	53.8	56.4
A2	9.3	6.9	6.7	69.2	34.8	43.2
A3	9.9	10.0	9.9	99.2	58.0	55.6
A4	9.9*	10.1*	9.6	84.8*	51.8*	64.0
A5	9.7**	9.2**	8.4	70.4**	25.6**	22.2
A6	9.4	7.5	8.2	85.4	56.8	62.2
A7	9.4**	7.7**	7.3	97.6**	82.4**	83.8
A8	7.7	7.2	7.0	75.2	37.4	52.5
A9	6.9	5.7	6.1	84.7	70.2	71.1
A10	9.6***	9.5***	9.5	96.5***	88.2***	76.5
N1	9.1	8.2	8.8	90.0	79.6	82.2
N2	8.2	7.5	7.5	90.0	59.0	64.6
P1	9.3**	8.1**	7.5	84.2**	0**	0.2

\* 16 week data

\*\* 8 week data

\*\*\* 4 week data

The surfactants which pass both the pH and peroxide stability tests are listed below (this eliminates the surfactants for which good peroxide stability is due predominantly to a decrease in composition pH). A3 and A4 are inferior in terms of pH stability because the pH has drifted upwards for them. In practice an upward drift may not be a problem because the benefits of an alkaline composition are retained.

In descending order of pH stability: A1 > A4/A3 > N1/A6

In descending order of peroxide stability: N1 > A6 > A3 > A4 > A1

Considering both pH and peroxide stability, the preferred surfactants are therefore those listed above. Especially preferred for their combination of pH stability and peroxide stability are the secondary alkane sulphonates A3 and A4.

5

In addition to pH and peroxide stability, phase behaviour must also be suitable. We screened the surfactants which passed the pH and peroxide stability criteria and obtained the following results in a procedure which is referred to in this specification as Test B. Test B consists of measuring the viscosity of a series of surfactant solutions with concentrations increasing by increments of 5%. The concentration above which the viscosity exceeds a value of 1200 cps is referred to in this specification as the threshold concentration. The test uses 1200 cps as being an approximate upper limit in terms of consumer acceptability. The level at which a particular surfactant is used in a liquid detergent should be below the threshold concentration, preferably by at least 5%, and more preferably by at least 9%. This is to allow for inaccurate dosing in the commercial manufacture of a liquid detergent and/or evaporation on storage. The results of the determination of the threshold concentrations for the preferred surfactants is given in Table 3. The numbers in brackets are the viscosity in centipoise at 25°C, 21s<sup>-1</sup> of the aqueous solution of surfactant at the specified concentration.

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**Table 3 - Results of Test B**

Surfactant	Concentration (wt.%) below which viscosity is < 1200 cp at 25°C, 21 s <sup>-1</sup>
<b>Anionic</b>	
A1	25 (1165)
A3	35 (925)
A4	40 (755)
A6	25 (15)
<b>Nonionic</b>	
N1	25 (675)

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The invention will now be further described with reference to the following non-limiting examples:

Table 4 gives details of the example formulations. All composition data is on a 100% w/w basis.

Table 4

Example:	1	2	3	4	5
Ingredient					
N1	14.0	17.5	7.5	7.5	15.4
A1	6.0	-	-	-	5.6
A3	-	7.5	17.5	-	7.0
A4	-	-		17.5	-
NaOH*	0.77	-	-	-	0.71
Dequest 2066	0.5				
DPA	0.06				
PVP	0.5				
Tinopal CBS-X	0.2				
Peroxide	5.0				
Water	to 100				
Initial pH	9.50	9.48	9.50	9.51	9.45
Viscosity (21 s <sup>-1</sup> , 25°C)	840	280	115	70	370

\*further NaOH is added to make the final pH adjustment

Formulations included PVP (polyvinylpyrrolidone) as an anti-redeposition and anti-dye transfer agent and TINOPAL CBS-X, an optical brightener ex Ciba Geigy. Storage Data for the formulations at a range of isothermal storage conditions is given in Tables 5 to 9. Both pH and residual peroxide data is given. Often, because peroxide is more stable under more acid conditions, a reasonable peroxide stability is obtained if there is a decrease in composition pH. A pH drop is undesirable because it reduces the detergency and it makes it harder to activate the peroxide with a bleach activator such as TAED.

**Table 5 - Example 1**

Time (weeks)	pH				Residual Peroxide (%)			
	5°C	25°C	37°C	45°C	5°C	25°C	37°C	45°C
0	9.50	9.50	9.50	9.50	100	100	100	100
1	9.39	9.25	9.22	9.16	-	-	-	-
2	9.63	9.37	9.37	9.24	100	99.59	96.69	96.48
4	9.34	9.22	9.19	8.98	-	-	-	-
8	9.37	9.07	8.92	7.21	100	97.52	88.61	82.61
12	9.43	9.13	8.73	7.0	100	94.21	84.89	77.64
16	9.30	9.13	7.77	6.50	100	94.20	84.88	81.98
24	9.47	9.03	7.47	6.05	100	92.55	78.70	75.80

**Table 6 - Example 2**

Time (weeks)	pH				Residual Peroxide (%)			
	5°C	25°C	37°C	45°C	5°C	25°C	37°C	45°C
0	9.48	9.48	9.48	9.48	100	100	100	100
1	9.36	9.44	9.32	9.42	100	100	99.60	98.60
2	9.54	9.16	9.14	9.14	100	100	97.60	95.19
4	9.44	9.08	9.03	7.35	100	100	95.79	89.38
8	9.35	8.87	8.14	6.86	97	100	92.86	88.17
12	9.52	9.03	7.52	6.50	100	100	90.60	92.60

Table 7 - Example 3

Time (weeks)	pH				Residual Peroxide (%)			
	5°C	25°C	37°C	45°C	5°C	25°C	37°C	45°C
0	9.50	9.50	9.50	9.50	100	100	100	100
1	9.40	9.65	9.32	9.30	98.80	99.60	98.60	100
2	9.61	9.47	9.51	9.38	100	98.00	96.60	91.38
4	9.46	9.37	9.38	8.88	100	99.00	87.20	79.36
8	9.31	9.15	9.05	6.99	100	95	82.17	70.94
12	9.55	9.35	9.15	6.85	100	96.2	80.6	71.70

Table 8 - Example 4

Time (weeks)	pH				Residual Peroxide (%)			
	5°C	25°C	37°C	45°C	5°C	25°C	37°C	45°C
0	9.51	9.51	9.51	9.51	100	100	100	100
1	9.57	9.55	9.56	9.62	100	100	98.97	92.61
2	9.86	9.67	9.74	9.78	100	100	96.72	85.42
4	9.61	9.55	9.47	9.35	100	98.56	78.03	73.10
8	9.68	9.57	9.44	7.80	98.15	96.72	75.16	59.10
12	9.88	9.59	9.37	7.00	100	98.15	73.70	55.10

Table 9 - Example 5

Time (weeks)	pH				Residual Peroxide (%)			
	5°C	25°C	37°C	45°C	5°C	25°C	37°C	45°C
0	9.45	9.45	9.45	9.45	100	100	100	100
1	9.53	9.49	9.42	9.39	100	100	95.42	92.03
2	9.58	9.50	9.40	9.31	99.4	98.41	95.62	91.44
4	9.62	9.46	9.32	8.16	100	99.0	90.24	81.08
8	9.80	9.63	9.05	6.99	100	100	86.70	78.50

## CLAIMS

1. A concentrated alkaline isotropic fabric washing liquid detergent composition comprising  
5 at least 15% by weight of surfactant and further comprises at least 0.2% by weight  
hydrogen peroxide bleach and an optical brightener, characterised in that the composition  
is substantially free from hydrotropes.
2. A composition according to claim 1 in which the composition comprises at least 20% by  
10 weight of surfactant.
3. A concentrated alkaline isotropic fabric washing liquid detergent composition comprising  
at least 23% of surfactants and further comprising at least 0.2% hydrogen peroxide bleach,  
characterised in that the composition is substantially free from hydrotropes.  
15
4. A composition according to any preceding claim in which the surfactant comprises anionic  
and nonionic surfactant.
5. A composition according to any preceding claim which further comprises an anti-  
20 redeposition agent.
6. A composition according to any preceding claim which further comprises an anti-dye  
transfer agent.
- 25 7. A composition according to any one of claims 4 to 6 in which the nonionic surfactant  
comprises more than 5% by weight of the total composition.
8. A composition according to claim 7 which comprises at most 5% of a surfactant which does  
not satisfy the pH targets when tested according to Test A as described herein.  
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9. A composition according to claim 8 comprising at most 5% of a surfactant which does not



satisfy the residual peroxide targets when tested according to Test A as described herein.

10. A composition according to claim 6 wherein at least 95% of the surfactant comprises surfactants which are being used at a level below their threshold concentration as determined by Test B described herein.  
5
11. A composition according to claim 3 in which all of the surfactants used satisfy the pH targets and preferably also the peroxide stability targets of Test A and are used at a level below their threshold concentration as determined by Test B.  
10
12. A method of washing laundry with a wash liquor comprising adding to the wash liquor a composition according to any preceding claim together with a bleach activator, preferably TAED.
- 15 13. A method of pre-treating a laundry item comprising administering the composition according to any one of claims 1 to 11 to a selected area of the item, leaving it in contact with the item for a short treatment period and then washing the item.

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C11D3/39 C11D1/83 C11D3/42

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,A	WO 96 30483 A (WARWICK INT GROUP ;DAY MICHAEL JOHN (GB); WALSH CAREY JAMES (GB)) 3 October 1996 cited in the application see claims 1-5,8 see page 3, line 28 - line 30 see page 4, line 17 - line 20 see page 5, line 11 - line 31	1-11,13
A	EP 0 548 019 A (CIBA GEIGY AG) 23 June 1993 see claims 11,17,20 --- -/--	1,4,7-12

☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

11 September 1997

Date of mailing of the international search report

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 93 14183 A (PROCTER &amp; GAMBLE) 22 July 1993  see page 2, line 32 - page 3, line 11  see page 7, line 1 - line 8  see page 10, line 32 - page 11, line 4  see page 15, line 19 - line 24  see claims 1-4</p> <p>---</p>	1-4,7-9
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